

Contract No.: W912DQ-15-D-3013
Task Order No.: 0002

US Army Corps of Engineers Kansas City District

Final Bench Scale Treatability Study Work Plan

**LCP Chemicals, Inc. Superfund Site
Remedial Design
Linden, Union County, New Jersey**

January 24, 2017





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PROJECT: Contract No.: W912DQ-15-D-3013
Task Order No. 002
LCP Chemicals, Inc. Superfund Site
Remedial Design
Linden, New Jersey

SUBJECT: Final Bench Scale Treatability Study Work Plan

Dear Travis:

CDM Federal Programs Corporation (CDM Smith) is pleased to submit the Final Bench Scale Treatability Study Work Plan for the LCP Chemicals Inc. Superfund Site, located in Linden, New Jersey.

We are providing copies of this submittal as follows:

- Travis Young, USACE KC District – 1 hard copy and 2 electronic copies on CD
- Jon Gorin, USEPA Region 2 – 1 hard copy and 1 electronic copy on CD
- Edwin Barth, USEPA Office and Research Development – 1 hard copy

If you have any questions or comments concerning this submittal, please feel free to call me at (732) 590-4638.

Sincerely,

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Project Manager
CDM Federal Programs Corporation

cc: A. Rahmani, CDM Smith
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Table of Contents

List of Acronyms	iii
Section 1 Introduction	1-1
1.1 Site Description.....	1-1
1.2 Summary of the Record of Decision.....	1-1
1.3 Effectiveness of Elemental Sulfur and Reactive Sulfides	1-3
1.4 Objectives of the Bench Scale Treatability Study and Summary of Tests to be Conducted.....	1-4
1.5 Work Plan Organization	1-5
Section 2 Soil Sample Preparation and Analyses	2-1
2.1 Soil Target Elemental Mercury Concentrations.....	2-1
2.2 Soil Sample Collection and Compositing.....	2-1
2.3 Composite Soil Sample Analyses	2-2
Section 3 Addition of Stabilization Additives	3-1
3.1 Reactive Sulfide Comparison and Selection	3-1
3.2 Test 1: Elemental Sulfur and In Situ Auger Mixing.....	3-1
3.3 Test 2: Elemental Sulfur and Ball Mill Processing	3-2
3.4 Test 3: Calcium Polysulfide and In Situ Auger Mixing	3-2
3.5 Test 4: FerroBlack® and In Situ Auger Mixing.....	3-2
Section 4 Analysis of Elemental Mercury in the Stabilized Soil Mixtures	4-1
Section 5 Solidification Tests	5-1
Section 6 Additional Analyses.....	6-1
Section 7 Evaluation and Reporting	7-1
Section 8 References	8-1

Figures

Figure 1 Site Plan and Pilot Study Area

Appendices

Appendix A SOP 1-1 Soil Sample Preparation
Appendix B SOP 1-2 Soil Mixing with Additives
Appendix C SOP 1-3 Elemental Mercury Analysis in Solid Samples
Appendix D SOP 1-4 Synthetic Precipitation Leaching Procedure and Semi-Dynamic Leaching
 Procedure on Stabilized Soils
Appendix E SOP 1-5 Unconfined Compressive Strength (Pocket Penetrometer)
Appendix F Reactive Sulfide Additive Comparison

List of Acronyms

BR	Brooks Rand
CDM Smith	CDM Federal Programs Corporation
CLP	Contract Laboratory Program
CH ₃ Hg	Methyl mercury
DESA	Division of Environmental Science and Assessment
DTL	Denver Treatability Laboratory
HgO	mercuric oxide
Hg	mercury
HgSO ₄	mercuric sulfate
HgCl ₂	mercuric chloride
HgS	mercuric sulfide
HgSe	mercury selenide
HgAu	mercury-gold amalgam
KC	Kansas City
LCP	LCP Chemicals Inc.
MBS®	Molecular Bonding System
mg/kg	milligram per kilogram
PTW	principal treat waste
RCRA	Resource Conservation and Recovery Act
RD	Remedial Design
ROD	Record of Decision
rpm	revolutions per minute
Site	LCP Chemicals Inc. Superfund Site
SOP	standard operating procedure
SPLP	synthetic precipitation leaching procedure
TAL	Target Analyte List
USACE	United States Army Corps of Engineers
USEPA	United States Environmental Protection Agency

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Section 1

Introduction

Under the United States Army Corps of Engineers (USACE), Kansas City District (KC), Contract No. W912DQ-15-D-3013, Task Order No. 002, CDM Federal Programs Corporation (CDM Smith) has been tasked to support the USACE-KC and the United States Environmental Protection Agency (USEPA) in providing technical services, completing the pre-design investigation, bench scale investigation, pilot study, and remedial design (RD) at the LCP Chemicals Inc. Superfund Site (the Site) located in Linden, Union County, New Jersey. This Bench Scale Treatability Study Work Plan establishes the approach for the upcoming laboratory bench scale study required to formulate the work plan for the full scale pilot study to be conducted at the Site and to obtain design parameters for the final RD.

1.1 Site Description

The Site is located in an industrial area of the Tremley Point peninsula in Linden, Union County, New Jersey. In 1955, the General Aniline and Film Corporation constructed and began operating a chlor-alkali plant on the 26-acre property of the Site. Areas within the LCP site were leased to other companies for the operation of related manufacturing operations. In 1957, a western portion of the property was leased to Union Carbide Corporation to house a hydrogen plant operation that used byproducts of the chlorine production. That facility, known as the Linde Division hydrogen plant, operated until 1990. In addition, Kuehne Chemicals, Inc. leased an area on the northern portion of the property to manufacture sodium hypochlorite.

The chlor-alkali manufacturing operations ceased by 1985. The Hanlin Group, Inc. filed a petition under Chapter 11 of the bankruptcy code and liquidated its assets by 1994. As part of the bankruptcy, the Hanlin Group abandoned the LCP property; ownership reverted to the bankruptcy estate.

The Site was placed on the National Priorities List in 1998. In 1999, a potentially responsible party, ISP-ESI, and USEPA entered into an Administrative Order to perform a Remedial Investigation and Feasibility Study. A Record of Decision (ROD) (USEPA 2014) was filed for the Site in February 2014.

1.2 Summary of the Record of Decision

In accordance with the ROD, the selected remedy for soil, sediments, groundwater, and buildings at the Site includes the following components:

- Installation of a capping system to prevent direct contact with soils and exposure to mercury vapor
- Treatment of the soil containing visible elemental mercury by mixing it with sulfur to convert the mercury to mercuric sulfide

- Excavation and onsite disposal of sediments and marsh soils from the North Off-Site Ditch and the downstream portion of the South Branch Creek
- Restoration of impacted wetlands and excavated areas
- Controlled demolition of the Site's buildings, recycling of non-porous material, and placement of porous material under the cap
- Containment and collection of the overburden groundwater layer by a barrier wall and collection/disposal trench
- Groundwater monitoring

The ROD stated that pre-design studies will be conducted to determine the amount of elemental sulfur needed to convert elemental mercury to mercuric sulfide. This document, Bench Scale Treatability Study Work Plan, is the first part of the pre-design studies. These bench scale tests will be conducted at CDM Smith's Denver Treatability Laboratory (DTL) using contaminated soil collected from the Site. The overall purpose of the bench scale treatability study is to evaluate the effectiveness of sulfur and various other chemicals as specified herein to stabilize or convert elemental mercury to mercuric sulfide in soil with visible elemental mercury. The results of the bench scale treatability study will be used to formulate a work plan for the full scale pilot study. The full scale pilot study is the second part of the pre-design studies and will be conducted at the Site.

The ROD specifies the use of elemental sulfur in three different applications:

- **Capped Area:** The proposed cap consists of the following layers from top to bottom – soil layer (24 inches), impermeable geosynthetic membrane, and elemental sulfur layer (3 inches).
- **Building Debris:** Elemental sulfur is proposed to treat porous material with visible signs of elemental mercury; the treated porous material would be reduced in size and placed under the cap.
- **Principal Treat Waste (PTW):** Elemental sulfur is proposed to treat (stabilize) contaminated soil with visible elemental mercury by converting the elemental mercury to mercuric sulfide; specifically, the method of sulfur application is by in situ techniques (i.e., in situ stabilization). The Final Feasibility Study (Cornerstone 2013) assumed only use of elemental sulfur with a range of 5 to 50 percent sulfur.

This work plan only addresses the PTW, and no tests are proposed concerning treatment of building debris or mercury vapor capture methods as part of the proposed cap.

The ROD also states the following concerning treatment of the PTW:

- Stabilization would be accomplished by in situ mixing of elemental sulfur with PTW soil through the use of specialized mixing equipment (e.g., augers).
- The amount of elemental sulfur per volume of soil will be determined during the pre-design studies.

- The measure of success for the full scale stabilization remedy would be the effectiveness of converting the elemental mercury to mercuric sulfide.
- The primary goal of stabilization would be to convert the elemental mercury to mercuric sulfide.

Each of these items is addressed in this work plan and proposed tests.

1.3 Effectiveness of Elemental Sulfur and Reactive Sulfides

The effectiveness of elemental sulfur treatment to convert elemental mercury to mercuric sulfide was reviewed by CDM Smith, and the findings were summarized in the memorandum titled “Use of Elemental Sulfur at the LCP Chemicals Inc. Superfund Site” dated August 19, 2016 (CDM Smith 2016). The major conclusions of the review follow:

- Elemental sulfur alone using the proposed application processes in the feasibility study (in situ mixing with augers) will most likely not be effective in converting elemental mercury in the PTW-contaminated soils to mercuric sulfide. The following conditions are necessary to convert elemental mercury to mercuric sulfide using elemental sulfur:
 - **Mechanical energy.** Ball mills or high energy mixers have been demonstrated to convert elemental mercury to mercuric sulfide using elemental sulfur (Lopez et al. 2008). Such high energy mechanical equipment potentially would be applied in an ex situ treatment method.
 - **Elevated temperature.** This approach does not appear to be appropriate or cost-effective for in situ application. Potentially, heat could be applied in an ex situ treatment method. The ex situ technique using the ball mill is more implementable and cost-effective than heating.
- To enable in situ conversion of elemental mercury to mercuric sulfide, use of reactive sulfides appears to be the most effective treatment additive. Examples of reactive sulfides that could be applied in situ include calcium polysulfide, sodium sulfide, Molecular Bonding System (MBS©) (proprietary chemicals), and FerroBlack© (proprietary nanoscale iron sulfide).
- Elemental sulfur as a layer in the cap and use of elemental sulfur to treat construction debris most likely will not be effective without some modification to the additives or treatment method.

Based on the above findings, the following additives were proposed to be tested during the bench scale treatability study to determine their effectiveness in converting elemental mercury to mercury sulfide:

- Elemental sulfur and auger mixing (baseline application specified in the ROD).
- Elemental sulfur and ball mill processing (potential ex situ application).

- Reactive sulfides and in situ auger mixing. A comparison will be performed to select two or three reactive sulfides for bench scale testing. These may include calcium polysulfide, sodium sulfide, FerroBlack®, and other proprietary sulfide additives.
- Reactive sulfides, cement, bentonite, and in situ auger mixing (selected samples for evaluation of physical properties).

No tests were proposed concerning treatment of building debris or mercury vapor capture methods.

1.4 Objectives of the Bench Scale Treatability Study and Summary of Tests to be Conducted

As stated in the ROD, pre-design studies will be conducted to determine the amount of elemental sulfur needed to convert elemental mercury to mercuric sulfide. This bench scale treatability study is the first part of the pre-design study but has been expanded in scope based on the review of the effectiveness of elemental sulfur and other additives summarized in Section 1.3.

Soil from the Site contaminated with visible elemental mercury (PTW) will be collected for the evaluations. The following additives and application methods will be tested to determine their effectiveness in converting elemental mercury to mercuric sulfide:

- Elemental sulfur alone plus in situ auger mixing. This is the “baseline” additive and application specified in the ROD. This application will be tested in the DTL and is designated Test 1 in this work plan.
- Use of elemental sulfur in a ball mill. This potentially could be used ex situ. This application will be tested in the DTL and is designated Test 2 in this work plan.
- Use of reactive sulfide for in situ applications with auger mixing. An evaluation of the demonstrated effectiveness and use, implementability, and relative cost was performed to select two reactive sulfides to test during the bench scale treatability study. The additives evaluated included calcium polysulfide, sodium sulfide, FerroBlack® and MBS®. Based on the evaluations (see Section 3.1), calcium polysulfide and FerroBlack® were selected as the two reactive sulfides to be evaluated as part of the bench scale treatability studies. Application using calcium polysulfide will be tested in the DTL and is designated Test 3 in this work plan. Application using FerroBlack® will be tested in the DTL and is designated Test 4 in this work plan.

The above tests will evaluate the effectiveness to stabilize the elemental mercury (i.e., convert the elemental mercury to mercuric sulfide) in the contaminated soil with visible elemental mercury (PTW). Typically, solidification is also performed in conjunction with stabilization. Solidification is completed by adding cement for strength and bentonite for permeability reduction and facilitation of mixing during the auger in situ mixing process. This bench scale treatability study will also include some limited solidification tests using cement and bentonite to evaluate potential physical characteristics of the treated soil and the potential advantage of such characteristics during full-scale implementation.

1.5 Work Plan Organization

This work plan contains the following sections:

Section 1: Introduction – This section includes a brief description of the Site, a summary of the ROD, a summary of the previous evaluation of elemental sulfur and reactive sulfides, and overall objectives and summary of tests to be performed during the bench scale treatability study.

Section 2: Soil Sample Preparation and Analysis – This section describes the target concentrations of elemental mercury in the soil to be used for the bench scale treatability study. Sample collection in the field, compositing in the DTL, addition of elemental mercury (if needed), and composite soil analyses are also discussed.

Section 3: Addition of Stabilization Additives – This section summarizes the selection of different additives to be tested and provides details of the tests to be conducted during the bench scale study:

Section 4: Analysis of Elemental Mercury in Stabilized Soil Mixtures – This section summarizes the method for analysis of elemental mercury in the stabilized soil mixtures.

Section 5: Solidification Tests – This section describes the solidification tests to be conducted with cement and bentonite.

Section 6: Additional Analyses – This section provides the list of laboratory analyses to be conducted on the stabilized and solidified soil mixtures.

Section 7: Evaluation and Reporting – This section summarizes the evaluations to be conducted and subsequent reporting of the results and evaluations.

Section 8: References – This section lists the documents and literature referenced in the work plan.

Appendices: Appendices provide detailed standard operating procedures for soil preparation, soil mixing with additives, elemental mercury analyses, leaching tests and unconfined compressive strength by pocket penetrometer. In addition, details of the comparison of reactive sulfides are provided.

Section 2

Soil Sample Preparation and Analyses

2.1 Soil Target Elemental Mercury Concentrations

According to the ROD, only PTW (soils with visible elemental mercury) will be treated. Besides the elemental mercury observed on the ground surface, elemental mercury was observed at 31 locations (39 actual samples, Table 6-3, RI Report, Brown and Caldwell 2013b). Typically, samples were not submitted for mercury analysis when they contained visible elemental mercury. As a result, only 12 analytical results are available for samples with visual elemental mercury (see Appendix J-1, RI Report, Brown and Caldwell 2013b). Five of these samples had relatively low total mercury concentrations, ranging from 0.11 to 471 milligrams per kilogram (mg/kg). Three samples had medium mercury concentrations (741 to 1,920 mg/kg), with an average of 1,460 mg/kg. Four samples had high mercury concentrations (4,300 to 7,870 mg/kg), with an average of 5,942 mg/kg. Based on these reported mercury concentrations, soils with two elemental mercury concentrations will be used during the bench scale treatability studies. The target mercury concentrations for the two samples will be:

- High concentration sample: 6,000 mg/kg
- Medium concentration sample: 1,500 mg/kg

2.2 Soil Sample Collection and Compositing

Soil samples will be collected during the field investigation from the identified test/study area (Figure 1). Previous sampling in this area observed elemental mercury to depths of 17 feet. Samples will be collected from a minimum of two different locations in the test/study area to prepare two composite samples for testing at the DTL. Samples will be collected over the entire depth of observed elemental mercury during drilling. If elemental mercury is not observed, the soil field descriptions and field screening results from the mercury vapor analyzer will be used to determine the interval of sampling. Equal volumes will be collected from each depth interval to create the composite. The exact number of increments cannot be specified at this time. However, based on the description of the soil types in the study area and the number of tests and test procedures for the bench scale treatability study described in this work plan, approximately 23 kg of soil is needed for each of the two composite samples. Assuming an approximate density of 1.6 grams per cubic centimeter, the volume of soil needed for each composite will be approximately 3.8 gallons. To provide some excess soil, a 5-gallon bucket of soil will be collected from each location and shipped to the DTL.

As discussed, the bench scale treatability study will be conducted utilizing two composite samples obtained from the test/study area. The two composite soil samples will be created in the DTL using Standard Operating Procedure (SOP) 1-1. As provided in SOP 1-1, samples will be air dried, homogenized, composited, sieved, and split to create uniform soil mixtures. Subsamples of the composite samples will be obtained according to SOP 1-1 and analyzed for elemental mercury at the DTL according to SOP 1-3. The soil with the higher elemental mercury concentration will be

labeled SS-H (high), and the soil with the lower elemental mercury concentration will be labeled SS-M (medium). Based on the reported mercury concentrations summarized in Section 2.1, the target mercury concentrations for the composite samples will be:

- SS-H: 6,000 mg/kg
- SS-M: 1,500 mg/kg

If the mercury levels need to be augmented to achieve the target starting concentrations, elemental mercury will be added to the composite soils as described in SOP 1-1, Section 5.3, Mercury Addition.

2.3 Composite Soil Sample Analyses

Subsamples of each final composite sample will be collected in accordance with SOP 1-1 and analyzed for a variety of baseline characteristics in accordance with the Quality Assurance Project Plan (QAPP) (CDM Smith 2017). Superfund's Field and Analytical Services Teaming Advisory Committee process will be employed to procure laboratory services. Sample analyses will proceed via a combination of Tier 1/2 Division of Environmental Science and Assessment/Contract Laboratory Program (DESA/CLP) and Tier 4 (DTL, CDM Smith Boston Geotechnical Laboratory, and CDM Smith-subcontracted laboratories). Baseline characterization of the two soil samples will include the following:

- Chemical Characterization (two samples plus one duplicate)
 - Target Analyte List (TAL) metals: – DESA/CLP (Tier 1/2).
 - Mercury Speciation (Five fractions¹): To identify forms of mercury present and quantify elemental mercury conversion to HgS – CDM Smith subcontract lab (Tier 4).
 - Leachability: A modified synthetic precipitation leaching procedure (SPLP) will be conducted at the DTL in accordance with SOP 1-4. The generated leachate will be analyzed for the eight Resource Conservation and Recovery Act (RCRA) metals – DESA/CLP (Tier 1/2).
- Physical Characterization (two samples)
 - Moisture content – CDM Smith Boston Geotechnical Laboratory (Tier 4)
 - Grain size distribution – CDM Smith Boston Geotechnical Laboratory (Tier 4)
 - Atterberg Limits – CDM Smith Boston Geotechnical Laboratory (Tier 4)
 - Bulk density – CDM Smith Boston Geotechnical Laboratory (Tier 4)

¹ Five extracts (F1-F5) for mercury speciation will be performed in addition to gaseous elemental mercury (F0): F1, water soluble = HgCl₂, (HgO and HgSO₄); F2, weak-acid soluble = HgO (HgSO₄); F3, organo-complexed = Hg-humic acid complexes, CH₃Hg, Hg₂Cl₂; F4, strongly complexed and elemental = elemental Hg, Hg₂Cl₂; F5, mineral-bound = HgS, HgSe, HgAu. Note: species in "(---)" indicate minor species reporting to the fraction or reporting to multiple fractions.

Section 3

Addition of Stabilization Additives

3.1 Reactive Sulfide Comparison and Selection

As previously summarized in the memorandum titled “Use of Elemental Sulfur at the LCP Chemicals Inc. Superfund Site” dated August 19, 2016 (CDM Smith 2016), to enable in situ conversion of elemental mercury to mercuric sulfide, use of reactive sulfides appear to be the most effective treatment additive. A comparison of the following reactive sulfides (Appendix F) was utilized to aid in the selection of stabilization additives to be tested:

- Calcium polysulfide
- Sodium sulfide
- FerroBlack®
- Molecular Bonding System (MBS®) chemicals

Based on the demonstrated use and effectiveness, implementability, and relative cost, the two additives selected were calcium polysulfide and FerroBlack® (Appendix F). Therefore, the following combinations of additives and application methods will be tested:

- Test 1: Elemental sulfur and in situ auger mixing
- Test 2: Elemental sulfur and ex situ ball mill processing
- Test 3: Calcium polysulfide and in situ auger mixing
- Test 4: FerroBlack® and in situ auger mixing

Both composite samples (SS-H and SS-M) will be tested using two or three concentrations of the additives (see following sections). Overall, 25 individual combinations of additives and soil will be tested. In addition, four duplicates (one from each additive type) and two blanks (soil without additive addition) will be tested (total of 31 samples). Details of the addition of the additives, concentrations of the additives, and processing methods are provided in SOP 1-2, Soil Mixing with Additives. Each of the combinations and applications is summarized in the following paragraphs.

3.2 Test 1: Elemental Sulfur and In Situ Auger Mixing

As previously discussed, the Final Feasibility Study (Cornerstone 2013) assumed a range of elemental sulfur addition from 5 to 50 percent and mixing with augers for 90 minutes. For soil SS-H, elemental sulfur will be added at concentrations of 5 and 25 weight percent of the soil. Addition of 50 weight percent elemental sulfur was considered, but this concentration may form soluble mercury-sulfur complexes, and overall, the two tested concentrations should provide adequate information to evaluate the effectiveness of elemental sulfur with the higher concentrations of elemental mercury. For soil SS-M, elemental sulfur will be added at

concentrations of 5, 12.5, and 25 weight percent. The soil/sulfur mixtures will be mixed in a standard paddle mixer at 10 to 15 revolutions per minute (rpm) for 90 minutes. This process best represents the in situ mixing with larger diameter augers.

3.3 Test 2: Elemental Sulfur and Ball Mill Processing

Based on review of literature (specifically Lopez et al. 2008), elemental sulfur should be added to the composite soils in a 1:1 weight ratio of the concentration of elemental mercury in the soil (i.e., 1,500 and 6,000 mg/kg of elemental sulfur would be added to SS-M and SS-H, respectively). This represents an excess sulfur content of 6.25 times the stoichiometric ratio based on the conversion of elemental mercury to mercuric sulfide (HgS). To provide a range of sulfur addition, elemental sulfur will be added to SS-M at concentrations of 1,000, 1,500, and 2,000 mg/kg. Elemental sulfur will be added to SS-H at concentrations of 4,000, 6,000, and 8,000 mg/kg. Concentrations of the elemental sulfur using ball mill processing are lower than concentrations of elemental sulfur proposed for use with auger mixing (Test 1). Less elemental sulfur is required during ball mill processing because of the large amount of mechanical energy introduced to convert elemental mercury to mercury sulfide (see Section 1.3). Very little mechanical energy is introduced during auger mixing; therefore, much higher concentrations of elemental sulfur are anticipated to be needed. Based on the literature (Lopez et al. 2008), elemental sulfur and mercury were processed in a planetary ball mill at approximately 400 rpm. For full scale production, rotary ball mills are used instead of planetary ball mills. Therefore, a rotary ball mill (sometimes called a jar mill) will be used for the bench scale tests. The critical rotational speed is based on literature formulas for grinding (Sahoo and Roy 2008) and was calculated to be between approximately 50 and 80 rpm, depending upon the diameter of the bench scale rotary vessel (jar). The mixing time will be 90 minutes.

3.4 Test 3: Calcium Polysulfide and In Situ Auger Mixing

Calcium polysulfide was selected as the first reactive sulfide to test and will be obtained from a commercial vendor (e.g., Calmet, Graus Chemicals). Calcium polysulfide is commercially available as a 29 weight percent solution. Based on the mercury concentrations and the bench and full scale tests conducted at the Mercury Refining Superfund Site in Colonie, New York (Brown and Caldwell 2013a), calcium polysulfide will be added to SS-M at concentrations of 1.5, 3, and 5 weight percent (weight percent based on mass of commercial product as purchased relative to the mass of soil) and to SS-H at 2, 6, and 10 weight percent in accordance with SOP 1-2. The mixtures will be processed to simulate in situ mixing with larger diameter augers (mixed in paddle mixer at 10 to 15 rpm for 90 minutes).

3.5 Test 4: FerroBlack® and In Situ Auger Mixing

FerroBlack® was selected as the second reactive sulfide to test and will be provided by the supplier REDOX Solutions (Carmel, Indiana). FerroBlack® will be obtained directly from the vendor before testing and typically should be used within several months of manufacture. Based on the recommendations of REDOX Solutions, stabilization with FerroBlack® will be tested with and without the addition of calcium oxide. For samples tested with calcium oxide, 10 weight percent (final mixture is 10 percent calcium oxide and 90 percent soil by weight) will be added to each sample and mixed using a paddle mixer. Once a temperature of 90 degrees Fahrenheit has

been reached, FerroBlack® will then be added to SS-M at a concentration of 5 weight percent (mixture is 5 percent FerroBlack® and 95 percent soil by weight, not counting the 10 percent calcium oxide) and to SS-H at a concentration of 10 weight percent in accordance with SOP 1-2. For samples tested without calcium oxide, FerroBlack® will be added to SS-M at concentrations of 2, 5, and 10 weight percent and to SS-H at 5, 10, and 15 weight percent. The mixtures will be processed to simulate in situ mixing with larger diameter augers (mixed in paddle mixer at 10 to 15 rpm for 90 minutes). Both the calcium oxide and the FerroBlack® will be mixed using the paddle mixer. As discussed above, the calcium oxide would be added and mixed first followed by the addition of FerroBlack®. This will represent full scale implementation during which calcium oxide would be added during the first pass of the auger and FerroBlack® during subsequent passes (i.e., a pass is one down and up mixing of the auger to complete depth).

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Section 4

Analysis of Elemental Mercury in the Stabilized Soil Mixtures

As previously discussed, the main performance criteria will be the conversion of the elemental mercury to mercuric sulfide. Each of the 31 mixtures discussed above will be analyzed for elemental mercury at the DTL using the procedure detailed in SOP 1-3, Analysis of Elemental Mercury in Solid Samples. An Ohio Lumex RA-915M Mercury Analyzer (Zeeman atomic adsorption spectrometry) with a Pyro-915+ attachment will be used to analyze each mixture for elemental mercury. The Pyro-915+ attachment will heat the soil samples to 150 to 210 degrees Celsius. At this temperature, the RA-915M will detect elemental mercury but not HgS. The concentration of elemental mercury in the stabilized samples will be compared to the initial concentrations (as measured by SOP 1-3) and a percent decrease calculated. This percent decrease will be assumed to be the percent of conversion of the elemental mercury to mercuric sulfide and will be used as the primary performance criteria for evaluation of effectiveness of each mixture. Either red cinnabar or black meta cinnabar forms are generated and are acceptable products. Additional observations and analyses of the various forms of mercuric sulfide will be made in accordance with SOP 1-3.

Section 6 of this work plan provides a description of additional laboratory analyses to be performed on up to 8 of the 25 stabilized soil mixtures. Laboratory analyses will be performed for mercury in accordance with the QAPP at DESA/CLP (Tier 1/2). Both SOP 3-1 and the DESA/CLP methods use an atomic adsorption technique at a wave length of 254 nanometers; however, the methods are not completely comparable due to differences in sample preparation procedures. The DESA/CLP methods use a chemical digestion, and SOP 3-1 uses thermal decomposition. Results from these two analytical procedures will be compared; however, an exact correlation is not anticipated because of the different preparation methods. In addition, the eight stabilized soil mixtures selected for additional analysis will be analyzed by a sub-contract laboratory (Tier 4) for mercury speciation via a sequential extraction method. Five forms of mercury will be reported in addition to gaseous elemental mercury. Fraction F5 will correspond to mercury sulfide (see previous footnote in Section 2.3).

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Section 5

Solidification Tests

Based upon the evaluation of the conversion of elemental mercury to mercuric sulfide, up to six of the most effective mixtures (conversion to mercuric sulfide using in situ techniques) will be selected for additional solidification tests in accordance with SOP 1-2. New splits of composite soil samples (SS-M and SS-H) will be stabilized and solidified to create new samples by adding the selected stabilizers and between 5 and 15 percent cement and between 1 and 3 percent bentonite (weight percentages based on mass of soil). The amounts of cement and bentonite are estimates, and the actual amounts will be determined in the DTL based on physical observations and strength. The stabilized and solidified mixtures will be placed in 2- x 3-inch plastic cylinders and let cure for a minimum of 7 days. The unconfined compressive strength will be measured at the DTL using a pocket penetrometer in accordance with SOP 1-5. In addition, the stabilized and solidified samples will be analyzed for elemental mercury in the DTL using SOP 3-1.

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Section 6

Additional Analyses

Based on the evaluation of the conversion of elemental mercury to mercuric sulfide (samples with the best conversion), up to 8 samples of the 25 stabilized mixtures (or the duplicates) will be analyzed for the following parameters in accordance with the QAPP (CDM Smith 2017):

- Chemical Characterization (Eight samples)
 - TAL metals: – DESA/CLP (Tier 1/2).
 - Mercury Speciation (Five fractions²): To identify the forms of mercury present and quantify elemental mercury conversion to HgS. – CDM Smith subcontracted lab (Tier 4).
 - Leachability: A modified SPLP will be conducted at the DTL in accordance with SOP 1-4. The generated leachate will be analyzed for the eight RCRA metals – DESA/CLP (Tier 1/2).
- Regulatory and Safety Evaluation (Eight samples)
 - pH (corrosivity) – CDM Smith DTL (Tier 4)
 - Reactivity (hydrogen sulfide gas generated at pH of 2) – CDM Smith DTL (Tier 4)

In addition, up to three of the six stabilized and solidified samples will be selected based on evaluation of the unconfined compressive strength measured at the DTL (SOP 1-5) and analyzed for the parameters below in accordance with the QAPP (CDM Smith 2017). The three samples selected will have a minimum unconfined compressive strength of 50 pounds per square inch and the highest effective conversion of elemental mercury to mercuric sulfide. The three samples will be analyzed for the following:

- Leachability (to evaluate any change due to the addition of cement and bentonite)
 - Leachability: A modified SPLP will be conducted at the DTL in accordance with SOP 1-4. The generated leachate will be analyzed for the eight RCRA metals – DESA/CLP (Tier 1/2).
 - Leachability: A semi-dynamic leach test will be conducted at the DTL in accordance with SOP 1-4. The generated leachate will be analyzed for mercury only – DESA/CLP (Tier 1/2).

² Five extracts (F1-F5) for mercury speciation will be performed in addition to gaseous elemental mercury (F0): F1, water soluble = HgCl₂, (HgO and HgSO₄); F2, weak-acid soluble = HgO (HgSO₄); F3, organo-complexed = Hg-humic acid complexes, CH₃Hg, Hg₂Cl₂; F4, strongly complexed and elemental = elemental Hg, Hg₂Cl₂; F5, mineral-bound = HgS, HgSe, HgAu. Note: species in “(---)” indicate minor species reporting to the fraction or reporting to multiple fractions.

- Physical Characterization
 - Unconfined compressive strength – CDM Smith Boston Geotechnical Laboratory (Tier 4)
 - Hydraulic conductivity – CDM Smith Boston Geotechnical Laboratory (Tier 4)
 - Moisture content – CDM Smith Boston Geotechnical Laboratory (Tier 4)
 - Bulk density – CDM Smith Boston Geotechnical Laboratory (Tier 4)

Section 7

Evaluation and Reporting

The results of all tests will be provided in the bench scale treatability study report. This report will include the results of all tests conducted at the CDM Smith DTL, CDM Smith Boston Geotechnical Laboratory, DESA/CLP, and the subcontract laboratories. The report will also provide an evaluation of the effectiveness of the tested additives to stabilize the elemental mercury in the composite soil samples based on the conversion of elemental mercury to mercuric sulfide. The report will evaluate the leachability and safety of the stabilized soil.

In addition, the report will include a limited evaluation of the leachability, strength, and hydraulic conductivity of the stabilized and solidified samples.

Finally, recommendations for the full-scale pilot study will be included in the report.

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Section 8

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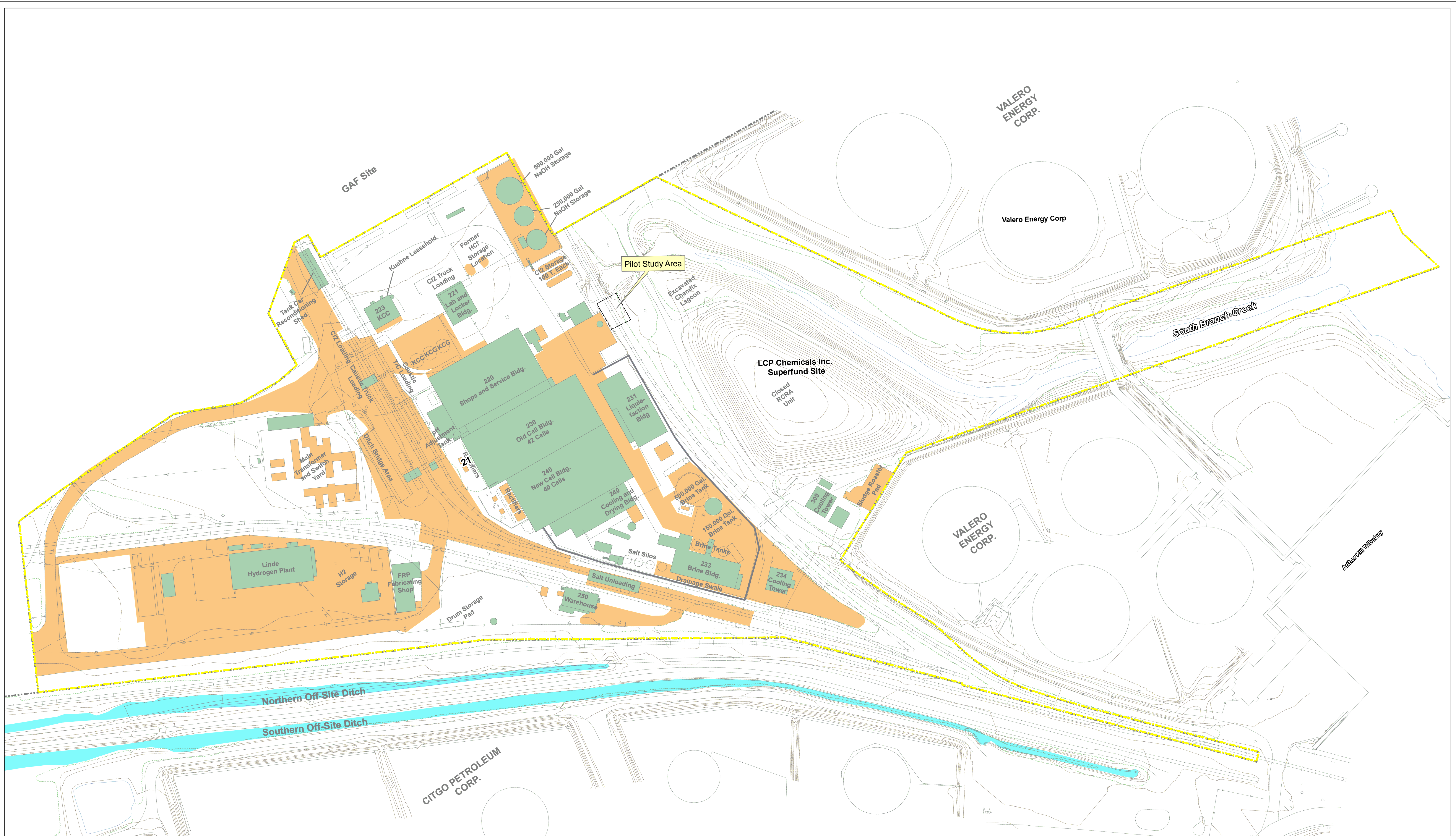
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Figures



Legend

--- Site Boundary

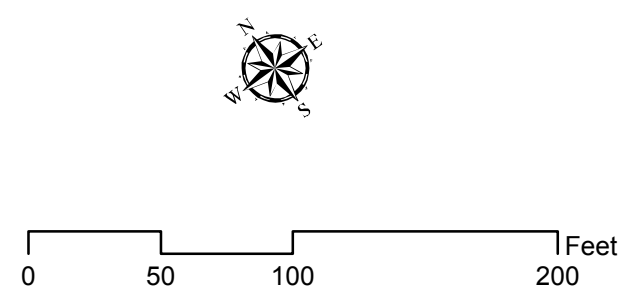


Figure 1
Site Plan and Pilot Study Area
LCP Chemicals Inc.,
Linden, NJ

Appendix A

Appendix A

SOP 1-1

Soil Sample Preparation

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Soil Sample Preparation

Project-Specific SOP 1-1
Revision: 1
Date: January 17, 2017

Prepared: Todd Burgesser
PM Approval: Thomas Mathew
LCP Chemicals Inc., Superfund
Project Name: Site. Mercury Stabilization Study

Technical Review: Roger Olsen
Editorial Review: Traci Mordell

1.0 Objective

This technical standard operating procedure (SOP) describes the laboratory procedures that will be followed to prepare contaminated soils for use in the stabilization/solidification bench scale treatability studies. This procedure includes soil drying, sieving and splitting/compositing, and the addition of elemental mercury (if needed). All procedures will be performed in the CDM Smith Denver Treatability Laboratory (DTL). Soil samples will be collected during the field investigation from the pilot study area. Samples will be collected from a minimum of two different locations to obtain the necessary quantities. After preparation and compositing of the two samples according to Section 5 of this SOP, subsamples will be analyzed for elemental mercury according to SOP 1-3, Elemental Mercury Analysis in Solid Samples. The soil with the higher elemental mercury concentration will be labeled SS-H (high), and the soil with the lower elemental mercury concentration will be labeled SS-M (medium). The target elemental mercury concentration for the SS-H sample will be 6,000 milligrams per kilogram (mg/kg), and the target concentration for the SS-M sample will 1,500 mg/kg. If the mercury levels need to be augmented to achieve the target starting concentrations, elemental mercury will be added as described in Section 5.3, Mercury Addition.

2.0 Background

The LCP Chemicals Inc. Superfund site is located in an industrial area of the Tremley Point peninsula in Linden, Union County, New Jersey. A chlor-alkali plant operated on the 26-acre property from 1955 to 1985. In 1991, the company filed for bankruptcy, and the property reverted to the bankruptcy estate. Soil and sediment are contaminated with various forms of mercury, including elemental mercury, in the vicinity of the mercury cell building. The Record of Decision (ROD February 2014) includes treatment of soil containing visible elemental mercury (principal threat waste [PTW]) by mixing it with sulfur to convert it to mercuric sulfide (i.e., stabilization). This SOP addresses the preparation of the soil for bench scale tests. Details of the additives (stabilization agents) to be tested are provided in other SOPs.

2.1 General Terms and Definitions

2.2 Associated Procedures

- SOP 1-3 Elemental Mercury Analysis in Solid Samples

3.0 General Responsibilities

Laboratory Manager – The laboratory manager is responsible for ensuring that laboratory personnel are trained in the use of this procedure, the required equipment, and health and safety procedures and that bedrock core samples are prepared in accordance with this procedure and any other SOPs pertaining to laboratory procedures. The laboratory manager must also ensure that the quantity and type of quality assurance (QA) samples collected meet the requirements of the work plans.

4.0 Project Planning

This section provides a list of general equipment used during soil preparation and various health and safety considerations.

4.1 General Equipment

- Site-specific plans (e.g., sampling, work, health and safety)
- Laboratory logbook
- Indelible black ink pens and markers
- Appropriate sample containers
- Labels and appropriate forms/documentation for sample shipment
- Plastic zip-top bags
- Personal protective clothing and equipment
- Teflon®-lined spatulas and pans and knives, trays, bowls, trowels, or spoons
- Decontamination supplies

Soil Sample Preparation

Project-Specific SOP 1-1

Revision: 1

Date: January 17, 2017

- Nitrile or appropriate gloves
- Sample containers
- Ice/Refrigerators
- 2 mil plastic sheeting
- Disposal spatulas, spoons, etc.
- Teflon®-lined baking pan
- Sample chain-of-custody forms
- Laboratory grade oven capable of 100°C +/- 2°C
- ¼" riffle splitter with catch pans (epoxy coated)
- Rotary tumbler
- Plastic sieves (U.S. sieve #10) and pan

5.0 Preparation Procedure

Upon receipt of contaminated soil samples from the field investigation, the samples will be unpacked from the cooler and checked against the chain-of-custody form. All procedures in this SOP will be performed in a fume hood.

5.1 Soil Drying

1. Transfer the contaminated soil to a tared plastic bowl and weigh to the nearest 0.1 gram (g). Record the mass.
2. Remove rocks (larger than approximately ¼ inch) and debris (e.g., wood, grass, glass). Reweigh.
3. Transfer the sample from the plastic bowl to an 18" x 26" x 1" Teflon®-lined baking sheet. Using a disposable plastic scoop, mix the sample. Place the pan containing the sample on the top shelf of the drying cart. Continue to mix the sample every hour to facilitate air drying.
4. After 24 hours, test the sample to ensure that the soil is dry enough to pass through the sieve. With a gloved hand, place a portion of the soil sample between the index finger and thumb. If the soil is dry, the soil will no longer stick together. Continue to allow the sample to air dry until the material is no longer cohesive.
5. Disaggregate any larger sized soil clods using a pestle with rubber end.
6. Once dry, transfer the soil to a plastic bowl and re-weigh the material. Record the mass to the nearest 0.1 g.
7. Transfer the soil material to a 1-gallon re-sealable baggie.

5.2 Soil Sieving

1. Following sample drying, transfer the dried soil material to an 18" #10 U.S. Sieve (2 millimeters [mm]) plastic sieve with a catch pan and lid.
2. Vigorously shake the sieve to separate materials smaller than 2 mm from the larger particles. Remove the lid from the sieve and inspect the retained material. If soil is remaining on the sieve, gently press the soil through the sieve.
3. Transfer the material that passed through the sieve and the material that was retained in the sieve to separate decontaminated plastic bowls and weigh the materials to the nearest 0.1g. Record the mass retained and the mass passed through the sieve.
4. Transfer the materials to an appropriately sized glass jar.

5.3 Mercury Addition (If Necessary)

1. Subsamples obtained following Section 5.4 instructions will be analyzed for elemental mercury according to SOP 1-3. If it is determined that elemental mercury needs to be added to the soil sample to achieve the target concentrations, quantitatively measure out the required mass of elemental mercury to the nearest 1 mg into a 25-milliliter (mL) glass beaker. Record the mass.
2. Transfer the entire soil sample to be augmented to a 2-liter (L) glass jar.
3. Add the elemental mercury from step 1, cap the jar, and shake to start distributing the mercury throughout the soil.
4. Wrap the jar containing the augmented sample in bubble wrap and place the jar in rotary tumbler that rotates at 30 +/- 1 revolutions per minute. Rotate the sample for a minimum of 2 hours.
5. Repeat step 4 as needed (see Section 7.1)
6. Transfer to a 1-gallon re-sealable baggie.

5.4 Soil Compositing and Splitting

1. Mix the soil in the sealed baggies by inverting the sample bag for several minutes.
2. Set up the riffle splitter in the fume hood by placing two catch pans in the slots where the sample will pass to.
3. Transfer the sample to the hopper of the ¼" riffle splitter, ensuring the material is spread out evenly along the top of the riffle splitter. Ensure that the soil is spread out evenly in the hopper.
4. Record the mass of each split.
5. Select one of the split samples to continue splitting until the desired mass for the tests required is achieved.

Soil Sample Preparation

Project-Specific SOP 1-1

Revision: 1

Date: January 17, 2017

6. Once the desired mass is achieved, transfer the sample to the appropriate labeled container or baggie for future testing.
7. Return all unused material to the labeled re-sealable baggie and repeat the steps above to create another sub-sample.
8. Create seven subsamples of approximately 200 mg each for analyses (see Section 7, Quality Control).

6.0 Equipment Cleaning/Decontamination

Disposable equipment will be used when available. In addition, plastic, Teflon, epoxy-coated, or Teflon-coated equipment will be used to prevent mercury amalgamation with metal components. To ensure that samples are not contaminated by equipment or containers, it is necessary to follow certain procedures for cleaning or decontaminating equipment. All equipment in direct contact with the sample must be cleaned between each sample preparation. Decontamination procedures for this equipment are discussed below.

6.1 Riffle Splitter, Sieves, Catch Pans, and Drying Pans

1. After each sample, use the dedicated vacuum to remove any loose ground material from the catch pans hopper and all other surfaces of the riffle splitter, sieves, catch pans, or drying pans.
2. Wash the equipment with phosphate detergent and a stiff brush, being sure to remove gross contamination.
3. Rinse all surfaces of the equipment with de-ionized or distilled water.
4. Using a spray bottle, apply a layer of phosphate-free detergent to all surfaces.
5. Vigorously scrub all surfaces of equipment.
6. Rinse all surfaces again with de-ionized or distilled water until all detergent has been removed.
7. With the exception of the riffle splitter, place equipment in 10 percent hydrochloric acid bath and soak for 24 hours.
8. Soak equipment in de-ionized or distilled water for 24 hours.
9. Rinse equipment three times with de-ionized or distilled water.
10. Place the equipment in the drying rack. To accelerate drying, the equipment can be placed in the oven at 60°C until dry.

7.0 Quality Control

Quality control procedures will consist of the mercury analysis per SOP 1-3. These analyses will be performed on splits of the composited soil created following Section 5-4. Sand decontamination blanks also will be analyzed.

7.1 Laboratory Duplicates of Composite Samples

After sample compositing and the initial split, a minimum of seven aliquots (approximately 0.5 g) will be removed from each split sample and analyzed for mercury by SOP 1-3. Seven is the minimum number of samples typically used for a valid statistical evaluation. A standard deviation of these analyses will be calculated as described below:

$$\sigma = \sqrt{\frac{1}{n} \sum_{i=1}^n (x_i - \bar{x})^2}$$

Where: σ = standard deviation
 x_1 = each value of the data set
 \bar{x} = second sample value (laboratory duplicate)
 n = total number of data points
 Σ = sum

CV = coefficient of variation = $(\sigma/\text{mean concentration}) \times 100\%$

A calculated CV of less than 50 percent will be considered acceptable. If the CV is greater 50 percent, the samples will be re-mixed in the rotary tumbler (see Section 5.3), splits generated, and the analyses repeated.

Soil Sample Preparation

Project-Specific SOP 1-1
Revision: 1
Date: January 17, 2017

7.2 Sand Decontamination Blanks

A sand decontamination blank will be prepared by passing an aliquot of clean quartz sand through the sieves after decontamination procedures in the same manner as the samples. Sand blanks will be prepared at a rate of approximately 1 per 20 samples. Analyze the sand blanks per SOP 3-1.

8.0 Documentation

Bound laboratory logbooks shall be used for the maintenance of laboratory records. All aspects of sample preparation, such as sample observations, deviations to procedures, laboratory duplicate precision, decontamination, and calibrations, shall be documented in the laboratory logbooks. The logs documenting the preparation sequence shall be documented daily.

All entries in laboratory logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's activities. Corrections to logbook entries will be accomplished by a single cross out with the date and initials of the person making the entry. White out or correction tape is not permitted.

Appendix B

Appendix B

SOP 1-2

Soil Mixing with Additives

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Soil Mixing with Additives

Project-Specific SOP 1-2

Revision: 1

Date: January 16, 2017

Prepared: Todd Burgesser

Technical Review: Roger Olsen

PM Approval: Thomas Mathew
LCP Chemicals Inc., Superfund

Editorial Review: Traci Mordell

Project Name: Site. Mercury Stabilization Study

1.0 Objective

This technical standard operating procedure (SOP) describes the laboratory procedures that will be followed to mix prepared composite soils with the additives for further testing. This procedure includes mixing of the prepared composite soils with the various additives (elemental sulfur and reactive sulfides) in an effort to stabilize (convert) the elemental mercury to mercury sulfide (HgS). In addition, solidification tests are outlined at the end of this SOP. The solidification tests will incorporate the addition of cement and bentonite to selected stabilized mixtures. All procedures will be performed in the CDM Smith Denver Treatability Laboratory (DTL).

Individual homogenous splits of 300 to 500 grams (g) of each composite soil (SS-M and SS-H) will be prepared as described in SOP 1-1. The following table lists the stabilization formulations that will be prepared.

Soil	Stabilization Agent	Concentration of Stabilization Additives
SS-M	Sulfur	Low
SS-M	Sulfur	Medium
SS-M	Sulfur	High
SS-M	Sulfur (mixed in a ball mill)	Low
SS-M	Sulfur (mixed in a ball mill)	Medium
SS-M	Sulfur (mixed in a ball mill)	High
SS-M	Calcium Polysulfide	Low
SS-M	Calcium Polysulfide	Medium
SS-M	Calcium Polysulfide	High
SS-M	FerroBlack®	Low
SS-M	FerroBlack®	Medium
SS-M	FerroBlack®	High
SS-M	FerroBlack® with Calcium oxide	Medium
SSH	Sulfur	Low
SS-H	Sulfur	High
SS-H	Sulfur (mixed in a ball mill)	Low
SS-H	Sulfur (mixed in a ball mill)	Medium
SS-H	Sulfur (mixed in a ball mill)	High
SS-H	Calcium Polysulfide	Low
SS-H	Calcium Polysulfide	Medium
SS-H	Calcium Polysulfide	High
SS-H	FerroBlack®	Low
SS-H	FerroBlack®	Medium
SS-H	FerroBlack®	High
SS-H	FerroBlack® with Calcium oxide	Medium

Soil Mixing with Additives

Project-Specific SOP 1-2

Revision: 1

Date: January 16, 2017

In addition, four duplicates (one for each additive) and two blanks (no addition of additives) will be prepared (see Section 8). In all, 31 mixtures will be prepared (25 stabilization tests + 6 duplicates/blanks = 31).

2.0 Background

The LCP Chemicals Inc. Superfund site is located in an industrial area of the Tremley Point peninsula in Linden, Union County, New Jersey. A chlor-alkali plant operated on the 26-acre property from 1955 to 1985. In 1991, the company filed for bankruptcy, and the property reverted to the bankruptcy estate. Soil and sediment are contaminated with various forms of mercury, including elemental mercury, in the vicinity of the mercury cell building. The Record of Decision (ROD February 2014) includes treatment of soil containing visible elemental mercury (principal threat waste [PTW]) by mixing it with sulfur to convert it to mercuric sulfide (i.e., stabilization). This SOP addresses the preparation of the soil with amendments for bench scale tests. Details of the additives (stabilization agents) to be tested are provided in other SOPs.

2.1 General Terms and Definitions

2.2 Associated Procedures

- SOP 1-1 Soil Sample Preparation
- SOP 1-3 Elemental Mercury Analysis in Solid Samples

3.0 General Responsibilities

Laboratory Manager – The laboratory manager is responsible for ensuring that laboratory personnel are trained in the use of this procedure, the required equipment, and health and safety procedures and that bedrock core samples are prepared in accordance with this procedure and any other SOPs pertaining to laboratory procedures. The laboratory manager must also ensure that the quantity and type of quality assurance (QA) samples collected meet the requirements of the work plans.

4.0 Project Planning

This section provides a list of general equipment used during rock core preparation and various health and safety considerations.

4.1 General Equipment

- Site-specific plans (e.g., sampling, work, health and safety)
- Laboratory logbook
- Indelible black ink pens and markers
- Appropriate sample containers
- Labels and appropriate forms/documentation for sample shipment
- Nitrile or appropriate gloves
- Sample containers
- Ice/Refrigerators
- 2"x3" plastic cylinders with endcaps
- Disposal spatulas, spoons, etc.
- Six place paddle mixer
- Rotary ball mill soil grinder
- Plastic zip-top bags
- Personal protective clothing and equipment
- Teflon®-lined spatulas and pans and knives, trays, bowls, trowels, or spoons
- Decontamination supplies
- Sample chain-of-custody forms
- Laboratory grade oven capable of 160°C +/- 2°C
- ¼" riffle splitter (epoxy coated) with catch pans
- Plastic bowls
- Analytical balance (0.01 g accuracy)
- 250 milliliter (mL) PTFE bottles
- Mercury Vapor Analyzer

5.0 Amendment Mixing Procedure

After preparation of the soil and any addition of elemental mercury (SOP 1-1), the soils (SS-H and SS-M) will be split by passing the sample through a ¼" riffle splitter to approximately 300 to 500 g aliquots as described in SOP 1-1. Mercury emissions will be monitored using the Mercury Vapor Analyzer during all tests. Additive addition and mixing procedures as well as concentrations vary among the different additives and are described in individual sections below.

Soil Mixing with Additives

Project-Specific SOP 1-2

Revision: 1

Date: January 16, 2017

5.1 Elemental Sulfur

1. Label 5 250 mL PTFE bottles and 5 1 L beakers with the sample IDs listed in the table in Step 3.
2. Transfer the contaminated soil to the tared 1 L glass beakers and weigh to the nearest 0.1 g. Record the mass and volume..
3. Quantitatively add elemental sulfur to each soil in the labeled beakers in the amounts and concentrations listed below:

Soil	Sample ID	Approximate Hg Concentration (mg/kg)	Target Sulfur Concentration Based on the soil mass (%)	Sulfur Mass to Add (assuming 250 g soil) g
SS-M	SS-M-S-5	1500	5	12.5
SS-M	SS-M-S-12.5	1500	12.5	31.25
SS-M	SS-M-S-25	1500	25	62.5
SS-H	SS-H-S-5	6000	5	12.5
SS-H	SS-H-S-25	6000	25	62.5

4. Record the mass and volume.
5. Place each beaker on the 6 place paddle mixer and lower the paddles into the soil mixture. Start the paddle mixer on and set the rate to level 5 (approximately 10 to 15 revolutions per minute [rpm]). Allow the samples to mix for 90 minutes at room temperature.
6. After 90-minutes of mixing, transfer the amended soil to a labelled 250 mL wide mouth PTFE bottle.
7. Record the volume.
8. Proceed with sample elemental mercury analysis by SOP 1-3.

5.2 Elemental Sulfur mixed with a Ball Mill

1. Label 6 250 mL PTFE bottles or ball mill jars with caps with the sample IDs listed in the table in Step 3.
2. Weigh to the nearest 0.1 g. Record the mass and volume.
3. Quantitatively add elemental sulfur to the ball mill jars at the concentrations listed below:

Soil	Sample ID	Approximate Hg Concentration (mg/kg)	Target Sulfur Concentration (mg/kg)	Sulfur Mass to Add (assuming 250 g soil) (mg)
SS-M	SS-M-S-1000-Mill	1500	1000	250
SS-M	SS-M-S-1500-Mill	1500	1500	375
SS-M	SS-M-S-2000-Mill	1500	2000	500
SS-H	SS-H-S-4000-Mill	6000	4000	1000
SS-H	SS-H-S-6000-Mill	6000	6000	1500
SS-H	SS-H-S-8000-Mill	6000	8000	2000

4. Record the mass and volume
5. Add 3 acrylic balls to the ball mill jars.
6. Place the ball mill jar(s) onto the ball mill rollers. Turn the ball mill on at full power (50-80 rpm) and set the timer to 90 minutes. Record the rpms.
7. After milling, transfer the amended soil to a labeled 250 mL PTFE wide mouth bottle using a disposable plastic spoon.
8. Record the volume.
9. Proceed with sample mercury analysis by SOP 1-3.

Soil Mixing with Additives

Project-Specific SOP 1-2

Revision: 1

Date: January 16, 2017

5.3 Calcium Polysulfide

1. Label 6 250 mL PTFE bottles and 6 1 L beakers with the sample IDs listed in the table in Step 3.
2. Transfer the contaminated soil to the tared 1 L glass beakers and weigh to the nearest 0.1 g. Record the mass and volume.
3. Quantitatively add calcium polysulfide (29 percent) to each beaker at the concentration listed below:

Soil	Sample ID	Approximate Hg Concentration (mg/kg)	Calcium Polysulfide Concentration (%)	Calcium Polysulfide Mass to Add (assuming 250 g soil) (g)
SS-M	SS-M-CPx-1.5	1500	1.5	3.75
SS-M	SS-M-CPx-3	1500	3	7.5
SS-M	SS-M-CPx-5	1500	5	12.5
SS-H	SS-H-CPx-2	6000	2	5
SS-H	SS-H-CPx-6	6000	6	15
SS-H	SS-H-FB-10	6000	10	25

4. Record mass and volume
5. Place each beaker on the 6 place paddle mixer and lower the paddles into the soil mixture. Start the paddle mixer and set the rate to level 5 (approximately 10 to 15 rpm). Allow the samples to mix for 90 minutes at room temperature.
6. After 90 minutes of mixing, transfer the amended soil to a labeled 250 mL wide mouth PTFE bottle.
7. Record volume
8. Proceed with sample mercury analysis by SOP 1-3.

5.4 FerroBlack® and Calcium Oxide (Quick Lime)

1. Label 8 250 mL PTFE bottles and 8 1 L beakers with the sample IDs listed in the table in Step 6.
2. Transfer the contaminated soil to the tared 1 L glass beakers and weigh to the nearest 0.1 g. Record the mass and volume.
3. Quantitatively add calcium oxide (CaO) to 2 soil sample bottles (see table below, 10 weight percent) and mix with the paddle mixer to a temperature of 90°F and uniform mixture. Record mass and volume.
4. Measure the temperature of the soil in each jar with a calibrated thermometer. If the temperature is above 90°F proceed to Step 6; if the temperature is below 90°F, proceed to Step 5.
5. Add 10 mL of room temperature deionized (DI) water to the soil mixture, re-cap the bottle, and mix the bottle to hydrate the mixture. Measure the mixture temperature to ensure the mixture is above 90°F. Record mass and volume.
6. During steps 4 and 5, measure the concentration of Hg vapor above the mixing container. Note: at 90°F, the vapor pressure of elemental Hg is estimated to be 0.0037 mm Hg.
7. Quantitatively add FerroBlack® to each soil sample beaker at the concentrations listed below:

Soil	Sample ID	Approximate Hg Concentration (mg/kg)	FerroBlack® Concentration (%)	FerroBlack® Mass to Add (assuming 250 g soil) (g)	Calcium Oxide Mass to Add (assuming 250 g soil) (g)
SS-M	SS-M-FB-2	1500	2	5	0
SS-M	SS-M-FB-5	1500	5	12.5	0
SS-M	SS-M-FB-10	1500	10	25	0
SS-M	SS-M-FB-CaO-5	1500	5	12.5	25
SS-H	SS-H-FB-5	6000	5	12.5	0
SS-H	SS-H-FB-10	6000	10	25	0
SS-H	SS-H-FB-15	6000	15	37.5	0

Soil Mixing with Additives

Project-Specific SOP 1-2

Revision: 1

Date: January 16, 2017

SS-H	SS-H-FB-CaO-10	6000	10	25	25
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- Record mass and volume.
- Place each beaker on the 6 place paddle mixer and lower the paddles into the soil mixture. Start the paddle mixer on and set the rate to level 5 (approximately 10 to 15 rpm). Allow the samples to mix for 90 minutes at room temperature.
- After 90 minutes of mixing, transfer the amended soil to a labeled 250 mL wide mouth PTFE bottle.
- Record mass and volume.
- Proceed with sample mercury analysis by SOP 1-3.

6.0 Solidification Tests

Up to 6 solidification tests may be performed based on the results of the stabilization tests performed in Section 5. The most successful treatments for each soil (SS-M and SS-H) will be selected. After the stabilization additives have been added to the composite soils, a variety of concentrations of Portland cement and bentonite as solidification agents will be tested as described in the following table:

- Transfer the stabilized soil to the tared 1 L glass beakers and weigh to the nearest 0.1 g. Record the mass and volume.
- Quantitatively add Portland Cement and bentonite (masses indicated below) and stir the mixture with a plastic spoon for 1 minute.

Soil	Amended Soil Mass (g)	Portland Cement/Bentonite Target Concentration (%)	Portland Cement Mass to Add (g)	Bentonite Mass to Add (g)
SS-M	250	5/1	12.5	2.5
SS-M	250	10/2	25	5
SS-M	250	15/3	37.5	7.5
SS-H	250	5/1	12.5	2.5
SS-H	250	10/2	25	5
SS-H	250	15/3	37.5	7.5

- Record mass and volume
- Quantitatively add DI water to the mixture to hydrate the amended soil and mix for 5 minutes using the paddle mixer.
- Pack the hydrated amended soil mixture into a labeled 2"x3" plastic cylinder that have been capped on one end and pack the soil tightly.
- Once packed, cap the opened end of the cylinder, seal both ends with electrical tape, and allow the sample to cure for a minimum of 7 days.
- Record mass and volume after 7 days.
- After the sample has cured for 7 days, proceed with the pocket penetrometer test by SOP 1-5.

7.0 Equipment Cleaning/Decontamination

Disposable equipment will be used when available. In addition, plastic, Teflon, epoxy-coated, or Teflon-coated equipment will be used to prevent mercury amalgamation with metal components. To ensure that samples are not contaminated by equipment or containers, it is necessary to follow certain procedures for cleaning or decontaminating equipment. All equipment in direct contact with the sample must be cleaned between each sample. Decontamination procedures for this equipment are discussed below.

7.1 Pans, Mixing Bowls, Ball Mill Shell, and Spoons

- After each sample, use a dedicated vacuum to remove any loose material from all other surfaces.
- Wash the equipment with phosphate detergent and a stiff brush, being sure to remove gross contamination.
- Rinse all surfaces of the equipment with deionized or distilled water.

Soil Mixing with Additives

Project-Specific SOP 1-2

Revision: 1

Date: January 16, 2017

4. Using a spray bottle, apply a layer of phosphate-free detergent to all surfaces.
5. Vigorously scrub all surfaces of equipment.
6. Rinse all surfaces again with DI or distilled water until all detergent has been removed.
7. Place equipment (not stainless steel ball mill shells) in 10 percent hydrochloric acid bath and soak for 24 hours.
8. Soak equipment in DI or distilled water for 24 hours.
9. Rinse equipment three times with DI or distilled water.
10. Place the equipment in the drying rack. To accelerate drying, the equipment can be placed in the oven at 60°C until dry.

8.0 Quality Control

Quality control procedures will consist of the mercury analysis per SOP 1-3. These analyses will be performed on duplicates of the composited soil created following Section 5. Also soil blanks will be analyzed. Sand decontamination blanks also will be analyzed.

8.1 Laboratory Duplicates

Create a duplicate amended soil for each amendment. For example, one duplicate will be performed for the sulfur amendment, sulfur in a ball mill, calcium polysulfide, and FerroBlack® additives. The precision of each test will be determined by calculating the relative percent difference (RPD) between the replicates, by measuring the mass of elemental mercury converted to mercury sulfide during the stabilization test. RPD is calculated as follows:

$$RPD = \frac{[(A-B)/A+B] \times 100}{2}$$

Where: A = Original Sample Result
B = duplicate sample result

If the RPD is greater than +/- 50 percent, the samples will be re-prepared.

8.2 Soil Blanks

Both soil samples will be processed according to Section 5.1 but without the addition of sulfur. Analyze the soil samples per SOP 3-1.

8.3 Sand Decontamination Blanks

A sand decontamination blank will be prepared by passing an aliquot of clean quartz sand over reusable equipment, including bowls, beakers, and ball mill equipment. Sand blanks will be prepared at a rate of approximately one per amendments type. Analyze the sand blanks per SOP 3-1.

9.0 Documentation

Bound laboratory logbooks shall be used for the maintenance of laboratory records. All aspects of sample preparation, such as sample observations, deviations to procedures, laboratory duplicate precision, decontamination, and calibrations, shall be documented in the laboratory logbooks. The logs documenting the preparation sequence shall be documented daily.

All entries in laboratory logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's activities. Corrections to logbook entries will be accomplished by a single cross out with the date and initials of the person making the entry. White out or correction tape is not permitted.

Appendix C

Appendix C

SOP 1-3

Elemental Mercury Analysis in Solid Samples

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Elemental Mercury Analysis in Solid Samples

Project-Specific SOP 1-3

Revision: 1

Date: January 16, 2017

Prepared: Todd Burgesser

Technical Review: Roger Olsen

PM Approval: Thomas Mathew

Editorial Review: Traci Mordell

Project Name: LCP Chemicals Inc., Superfund
Site: Mercury Stabilization Study

1.0 Objective

This technical standard operating procedure (SOP) describes the laboratory procedures that will be followed to analyze prepared soils for mercury. The user manual for the instrumentation to be used (Ohio Lumex PYRO-915+ coupled to the RA-915M) should also be available. The RA-915M is a portable multifunctional atomic absorption analyzer. This instrument incorporates Zeeman Background correction, which eliminates the effects of potential interferences. The PYRO-915+ is an attachment that converts species of mercury into elemental mercury using thermal decomposition with subsequent transportation of released mercury vapor to the analytical cell (RA-915M). Multiple pre-programmed modes are available for use that limit the temperature of the thermal chamber. These modes allow the user to select the species of mercury that will be thermally decomposed and transported to the analyzer. For this study, elemental mercury is the species of interest, and Mode 8 will be selected. The decomposition temperature in the first chamber using Mode 8 is 150 to 210°C. All procedures will be performed in the CDM Smith Denver Treatability Laboratory (DTL).

Individual homogenous splits of up to 1 gram (g) of each soil to be analyzed will be prepared as described in SOP 1-1. Multiple duplicates will be prepared and analyzed to ensure that quality objectives are achieved. In addition to multiple duplicate analyses performed following these procedures, additional analyses at a contracted laboratory (DESA/CLP) will also be performed. Although a direct correlation of these methods (DESA/CLP and SOP 1-3) is not possible due to differences in sample preparation methods, these analyses will be compared qualitatively.

The results of the elemental mercury analyses will be used to evaluate the effectiveness of the various additives to convert elemental mercury to mercuric sulfide. Specifically, the elemental mercury concentrations of the stabilized samples will be compared to the initial elemental mercury concentrations in the composite soils and the percent decrease in elemental mercury calculated. This percent decrease will be assumed to be the amount of elemental mercury converted to mercuric sulfide.

2.0 Background

The LCP Chemicals Inc. Superfund site is located in an industrial area of the Tremley Point peninsula in Linden, Union County, New Jersey. A chlor-alkali plant operated on the 26-acre property from 1955 to 1985. In 1991, the company filed for bankruptcy, and the property reverted to the bankruptcy estate. Soil and sediment are contaminated with various forms of mercury, including elemental mercury, in the vicinity of the mercury cell building. The Record of Decision (ROD February 2014) includes treatment of soil containing visible elemental mercury (principal threat waste [PTW]) by mixing it with sulfur to convert it to mercuric sulfide (HgS) (i.e., stabilization). This SOP addresses the analysis of the soil and soil with amendments for mercury in bench scale tests.

2.1 General Terms and Definitions

2.2 Associated Procedures

- SOP 1-1 Soil Sample Preparation
- SOP 1-2 Soil Mixing with Additives

3.0 General Responsibilities

Laboratory Manager – The laboratory manager is responsible for ensuring that laboratory personnel are trained in the use of this procedure, the required equipment, and health and safety procedures and that bedrock core samples are prepared in accordance with this procedure and any other SOPs pertaining to laboratory procedures. The laboratory manager must also ensure that the quantity and type of quality assurance (QA) samples collected meet the requirements of the work plans.

4.0 Project Planning

This section provides a list of general equipment used during rock core preparation and various health and safety considerations.

4.1 General Equipment

- Site-specific plans (e.g., sampling, work, health and safety)
- Laboratory logbook
- Indelible black ink pens and markers
- Appropriate sample containers
- Labels and appropriate forms/documentation for sample shipment
- Nitrile or appropriate gloves
- Sample containers
- Disposal spatulas, spoons, etc.
- Ohio Lumex Pyro 915+
- Personal protective clothing and equipment
- Teflon®-lined spatulas and pans and knives, trays, bowls, trowels, or spoons
- Decontamination supplies
- Sample chain-of-custody forms
- Analytical balance (0.01 g accuracy)
- Ohio Lumex RA-915+ or RA-915M

5.0 Mercury Analysis and Quality Control

Prepared soils (SOP 1-1) and soils amended with stabilization chemicals will be analyzed for elemental mercury using a Ohio Lumex PYRO-915+ coupled to the RA-915M. The Pyro-915+ unit heats a portion of the prepared sample at 150 to 210°C to volatilize the elemental mercury. The mercury vapor is transported to the RA-915M where it is analyzed by Zeeman atomic adsorption spectrometry (ZAAS). Specific instrument operating instructions can be found in the appendix to this SOP. This appendix contains the user manuals for the Ohio Lumex PYRO-915+ and the RA-915M.

5.1 Instrument Setup

Upon receipt of the PYRO-915+ and the RA-915+, unpack the instruments and follow directions for instrument setup provided in the user manual.

5.2 Instrument Calibration and Method Validation

The mercury analysis system will be calibrated initially by analyzing a 5-point calibration curve. Development of the curve should be accomplished through the preparation of elemental mercury in soil standards. These standards should be prepared in certified clean soil or sand that has been ground and homogenized as described in SOP 2-1. Calibration concentration will range from 1 to 10,000 milligrams per kilogram (mg/kg) elemental mercury in soil. Calibrate the system in Mode 8 (150 to 210°C). The instrument is PC operated. The instrument includes complete calibration software that calculates best fit curves and provides statistics based on the correlation coefficient of the fitted curve. A fit of 0.995 R² or better for the initial calibration is required before soil analysis can proceed.

A continuing calibration standard (one of the initial calibration soil standards) will be analyzed at a minimum frequency of 1 per 20 samples. The accuracy of all continuing calibration verification samples must be between 50 and 150 percent of the standard. The continuing calibration standard should be in the midpoint of the calibration curve.

After initial calibration, validate the method to ensure that elemental mercury is the primary species of mercury measured using Mode 8. Prepare two soil standards as described above by using (1) black metacinnabar (beta-HgS) and (2) red cinnabar (alpha-HgS) at 100 mg/kg. Analyze this validation standard using Mode 8 and Mode 2 (520 to 580°C). In Mode 8, the mercury should not be detected. In Mode 2, mercury should be detected at approximately 100 mg/kg. If mercury is detected in Mode 8, the temperature range will be adjusted, and the validation procedure repeated. Selected stabilized materials will be analyzed in Mode 2 and Mode 8 in an effort to verify conversion of elemental mercury to HgS and to determine the form of HgS (black meta-cinnabar or red-cinnabar). Visual comparison of the product formed (black or red cinnabar) will also be documented.

5.3 Method Blanks

Elemental Mercury Analysis in Solid Samples

Project-Specific SOP 1-3

Revision: 1

Date: January 16, 2017

Analyze a method blank at a rate of 1 per 10 samples or 1 per day at a minimum. The method blank consists of certified mercury free silica sand. The mass of silica sand analyzed should be equivalent to the highest mass of calibration material analyzed during the initial calibration. Mercury should not be detected in the method blanks.

6.0 Equipment Cleaning/Decontamination

Disposable equipment will be used when available. In addition, plastic, Teflon, epoxy-coated, or Teflon-coated equipment will be used to prevent mercury amalgamation with metal components. To ensure that samples are not contaminated by equipment or containers, it is necessary to follow certain procedures for cleaning or decontaminating equipment. All equipment in direct contact with the sample must be cleaned between each sample. Decontamination procedures for this equipment are discussed below.

6.1 Scoops, Sample Boats, and Glassware

1. Wash the scoops, spoons, glassware, and other equipment that has contacted contaminated soil with phosphate free detergent and a stiff brush.
2. Rinse all surfaces of the equipment with de-ionized or distilled water three times.
3. Place equipment (not stainless steel ball mill shells) in 10 percent hydrochloric acid bath, soak for 24 hours.
4. Soak equipment in de-ionized or distilled water for 24 hours.
5. Rinse equipment three times with de-ionized or distilled water.
6. Place the equipment in the drying rack. To accelerate drying, the equipment can be placed in the oven at 60°C until dry.

7.0 Documentation

Bound laboratory logbooks shall be used for the maintenance of laboratory records. All aspects of sample preparation, such as sample observations, deviations to procedures, laboratory duplicate precision, decontamination, and calibrations, shall be documented in the laboratory logbooks. The logs documenting the preparation sequence shall be documented daily.

All entries in laboratory logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's activities. Corrections to logbook entries will be accomplished by a single cross out with the date and initials of the person making the entry. White out or correction tape is not permitted.

Appendix D

Appendix D

SOP 1-4

Synthetic Precipitation Leaching Procedure and
Semi-Dynamic Leaching Procedure on Stabilized
Soils

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Synthetic Precipitation Leaching Procedure and Semi-Dynamic Leaching Procedure on Stabilized Soils

Project-Specific SOP 1-4
Revision: 1
Date: January 16, 2017

Prepared: Todd Burgesser

Technical Review: Roger Olsen

PM Approval: Thomas Mathew
LCP Chemicals Inc., Superfund

Editorial Review: Traci Mordell

Project Name: Site. Mercury Stabilization Study

1.0 Objective

This technical standard operating procedure (SOP) describes the laboratory procedures that will be followed to prepare and leach composite soil and stabilized and solidified soils for leaching by the synthetic precipitation leaching procedure (SPLP) by modified EPA SW-846 1312 and the semi-dynamic leaching procedure (SDL) by modified EPA 1315 and ASTM 1308. SPLP will be performed on the two initial soil samples (SS-M and SS-H) prepared as described in SOP-1-1 as well as eight of the stabilized samples and three of the stabilized and solidified samples prepared as described in SOP 1-2. The SDL procedure will be performed on three of the stabilized and solidified samples from SOP 1-2.

The SPLP procedure will use extraction fluid #1 at a pH of 4.2 (SW-846 1312). The SPLP procedure will be modified to use a 2:1 liquid to solid ratio. The SDL procedure will be modified to incorporate nine sampling intervals at times contained in both ASTM 1308 and EPA 1315. These sampling intervals will be 2 hours, 24 hours, 48 hours, 72 hours, 7 days, 14 days, 21 days, 28 days, and 42 days. The solidified cylinders will be leached using SPLP extraction fluid #1 at a pH of 4.2. The liquid to surface area ratio will be maintained at approximately 10:1 milliliter per square centimeters. All leaching procedures will be performed in the CDM Smith Denver Treatability Laboratory (DTL). The leachate generated during the SPLP and the SDL procedures will be submitted to an analytical lab for analysis of total mercury.

2.0 Background

The LCP Chemicals Inc. Superfund site is located in an industrial area of the Tremley Point peninsula in Linden, Union County, New Jersey. A chlor-alkali plant operated on the 26-acre property from 1955 to 1985. In 1991, the company filed for bankruptcy, and the property reverted to the bankruptcy estate. Soil and sediment are contaminated with various forms of mercury, including elemental mercury, in the vicinity of the mercury cell building. The Record of Decision (ROD February 2014) includes treatment of soil containing visible elemental mercury (principal threat waste [PTW]) by mixing it with sulfur to convert it to mercuric sulfide (i.e., stabilization). This SOP addresses the leaching of stabilized and solidified soils. Details of the additives (stabilization agents) to be tested are provided in other SOPs.

2.1 General Terms and Definitions

2.2 Associated Procedures

- SOP 1-1 Soil Sample Preparation
- SOP 1-2 Soil Mixing with Additives

3.0 General Responsibilities

Laboratory Manager – The laboratory manager is responsible for ensuring that laboratory personnel are trained in the use of this procedure, the required equipment, and health and safety procedures and that bedrock core samples are prepared in accordance with this procedure and any other SOPs pertaining to laboratory procedures. The laboratory manager must also ensure that the quantity and type of quality assurance (QA) samples collected meet the requirements of the work plans.

4.0 Project Planning

This section provides a list of general equipment used during SDL and SPLP preparation and various health and safety considerations.

4.1 General Equipment

- Site-specific plans (e.g., sampling, work, health and safety)
- Laboratory logbook
- Plastic zip-top bags
- Personal protective clothing and equipment

Synthetic Precipitation Leaching Procedure and Semi-Dynamic Leaching Procedure on Stabilized Soils

Project-Specific SOP 1-4
Revision: 1
Date: January 16, 2017

- Indelible black ink pens and markers
- Appropriate sample containers
- Labels and appropriate forms/documentation for sample shipment
- Nitrile or appropriate gloves
- Sample containers
- Ice/Refrigerators
- 2"x3" plastic cylinders with endcaps
- Disposal spatulas, spoons, etc.
- 12" 2 millimeter (mm) stainless steel sieve
- Extraction Fluid #1
- Peristaltic pump
- pH meter with pH electrode and oxidation-reduction potential (ORP) electrode
- Teflon®-lined spatulas and pans and knives, trays, bowls, trowels, or spoons
- Decontamination supplies
- Sample chain-of-custody forms
- Laboratory grade oven capable of 160°C +/- 2°C
- ¼" riffle splitter (epoxy coated) with catch pans
- Plastic steel bowls
- Rotary tumbler
- Analytical balance (0.01 gram [g] accuracy)
- 500 milliliter (mL) PTFE bottles
- Silicon tubing
- Conductivity meter

5.0 Modified SPLP Procedure

After analysis of amended soils (SOP 1-3) with stabilization amendments and/or solidification amendments, up to eight stabilized soils and three solidified soils along with the two original non-treated soils will be disaggregated, sieved, and passed through a riffle splitter to obtain a representative for SPLP leaching. This procedure is described in SOP 1-1, Soil Sample Preparation.

5.1 SPLP Leaching

1. Label twelve 500 mL PTFE bottles with the relevant sample IDs of samples to be leached (2 original non-amended soils, 8 stabilized soils, and 3 solidified soils).
2. Transfer 25 g of the contaminated soil to the tared 500 mL PTFE bottles and weigh to the nearest 0.1 g. Record the mass.
3. Quantitatively add 50 mL of extraction fluid # 1 to each bottle. Record the exact volume added. Note: This will create a 2:1 (mL:g) ratio. This ratio is more representative of actual in situ leaching by groundwater than the standard SPLP ratio of 20:1.
4. Securely cap each bottle and invert the bottle to mix the soil and extraction fluid.
5. Uncap the bottles and measure the solution pH, ORP, and conductivity and record the measurements.
6. Cap the bottles and secure the cap with electrical tape.
7. Place each bottle in the rotary tumbler drum and pack the drum with bubble wrap to secure the bottles. Place the lid on the drum.
8. Rotate the bottles for 24 hours at 30 revolutions per minute (rpm).
9. After the 24-hours tumbling time, remove the sample bottles from the tumbler and allow them to sit for 30 minutes to settle the solids.
10. Remove the caps from the bottles and measure the pH, ORP, and conductivity and record the measurements.
11. Decant the solution into a labeled preserved sample bottle for analysis of total mercury.
12. Submit the samples to the contracted laboratory for total mercury analysis.

5.2 SDL Leaching

1. Label three 2000 mL leaching vessels (hermetic glass jar with lid and a rubber gasket preventing contact with the leaching fluid).
2. Measure the mass and dimensions of each unmolded cylinder. Each 2"x3" cylinder should have an approximate surface area of 200 square centimeters (cm²). Record the measurements and calculate the surface area.
3. Suspend each cylinder (mold removed) in the leaching vessel by constructing a sling out of Teflon disks (top and bottom) and Teflon string. Place the cylinder between the disks, and secure with the Teflon string. Attach the Teflon string to the outside of the vessel with packaging tape. The Teflon disks are designed in a way to contact the cylinder or core minimally at the very edges of the top and bottom of the cylinder. Suspend the cylinder at a minimum of 1 centimeter from the bottom and walls of the leaching vessel (glass jar) at all times. The Teflon string should not come into contact with the cylinder.
4. Quantitatively transfer the appropriate volume of extraction fluid #1 to the vessel. The volume of extraction fluid will equal the surface area of the cylinder times 10. Approximately 2000 ml.

Synthetic Precipitation Leaching Procedure and Semi-Dynamic Leaching Procedure on Stabilized Soils

Project-Specific SOP 1-4
Revision: 1
Date: January 16, 2017

5. At the specified sampling intervals (2 hours, 24 hours, 48 hours, 72 hours, 7 days, 14 days, 21 days, 28 days, and 42 days), open the leaching vessel and transfer the leachate to a clean labeled 500 ml nitric acid (HNO₃) preserved sample bottle and a 2 L glass beaker with a peristaltic pump and clean silicon tubing. Every attempt should be made not to touch or disturb the cylinder.
6. Measure the pH, ORP, and conductivity of the leachate contained in the 2 L beakers and record the measurements.
7. Submit the sample (500 ml HNO₃ preserved bottle) to the contracted laboratory for analysis of mercury.
8. Repeat Steps 4 through 7 for each sampling interval.

6.0 Equipment Cleaning/Decontamination

Disposable equipment will be used when available. In addition, plastic, Teflon, epoxy-coated, or Teflon-coated equipment will be used to prevent mercury amalgamation with metal components. To ensure that samples are not contaminated by equipment or containers, it is necessary to follow certain procedures for cleaning or decontaminating equipment. All equipment in direct contact with the sample must be cleaned between each sample. Decontamination procedures for this equipment are discussed below:

6.1 Beaker, Cylinder, and Leaching Vessels

1. Rinse all surfaces of the glassware with de-ionized or distilled water.
2. Using a spray bottle, apply a layer of phosphate-free detergent to all surfaces.
3. Vigorously scrub all surfaces of equipment.
4. Rinse all surfaces again with de-ionized or distilled water until all detergent has been removed. Perform in triplicate.
5. Place equipment in 10 percent hydrochloric acid bath, and soak for 24 hours.
7. Soak equipment in de-ionized or distilled water for 24 hours.
6. Rinse equipment three times with de-ionized or distilled water.
7. Place the equipment in the drying rack. To accelerate drying, the equipment can be place in the oven at 60°C until dry.

7.0 Quality Control

Quality control procedures will consist of the mercury analysis per SOP 1-3. These analyses will be performed on splits of the composited soil created following Section 5-4. Sand decontamination blanks also will be analyzed

7.1 Laboratory Duplicates

One duplicate sample from the SPLP leachate will be generated by splitting a leachate sample and sending it to the contract laboratory. The precision of each measurement will be determined by calculating the relative percent difference (RPD) between original sample result and the duplicate sample result. RPD is calculated as follows:

$$RPD = \frac{[(A-B)/A+B]}{2} \times 100$$

Where: A = Original Sample Result
B = duplicate sample result

Acceptable precision results should be +/- 20 percent RPD.

8.0 Documentation

Bound laboratory logbooks shall be used for the maintenance of laboratory records. All aspects of sample preparation, such as sample observations, deviations to procedures, laboratory duplicate precision, decontamination, and calibrations, shall be documented in the laboratory logbooks. The logs documenting the preparation sequence shall be documented daily.

All entries in laboratory logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's activities. Corrections to logbook entries will be accomplished by a single cross out with the date and initials of the person making the entry. White out or correction tape is not permitted.

Appendix E

Appendix E

SOP 1-5

Unconfined Compressive Strength (Pocket Penetrometer)

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Unconfined Compressive Strength (Pocket Penetrometer)

Project-Specific SOP 1-5
Revision: 1
Date: January 17, 2017

Prepared: Todd Burgesser

Technical Review: Roger Olsen

PM Approval: Thomas Mathew
LCP Chemicals Inc., Superfund

Editorial Review: Traci Mordell

Project Name: Site. Mercury Stabilization Study

1.0 Objective

This technical standard operating procedure (SOP) describes the laboratory procedures that will be followed to measure unconfined compressive strength using a pocket penetrometer. This procedure will be performed on soils that have been amended with stabilizing agents/additives and solidification agents/additives according to SOP 1-2. The solidification options in SOP 1-2 will incorporate the addition of a cement and bentonite mixture to selected mixtures. The cement and bentonite will be added to decrease the hydraulic conductivity of the soil mixture and to add additional strength. This procedure will be performed in the CDM Smith Denver Treatability Laboratory (DTL).

2.0 Background

The LCP Chemicals Inc. Superfund site is located in an industrial area of the Tremley Point peninsula in Linden, Union County, New Jersey. A chlor-alkali plant operated on the 26-acre property from 1955 to 1985. In 1991, the company filed for bankruptcy, and the property reverted to the bankruptcy estate. Soil and sediment are contaminated with various forms of mercury, including elemental mercury, in the vicinity of the mercury cell building. The Record of Decision (ROD February 2014) includes treatment of soil containing visible elemental mercury (principal threat waste [PTW]) by mixing it with sulfur to convert it to mercuric sulfide (i.e., stabilization). This SOP addresses the preparation of the soil with amendments for bench scale tests. Details of the additives (stabilization agents) to be tested are provided in other SOPs.

2.1 General Terms and Definitions

2.2 Associated Procedures

- SOP 1-2 Soil Mixing with Additives

3.0 General Responsibilities

Laboratory Manager – The laboratory manager is responsible for ensuring that laboratory personnel are trained in the use of this procedure, the required equipment, and health and safety procedures and that bedrock core samples are prepared in accordance with this procedure and any other SOPs pertaining to laboratory procedures. The laboratory manager must also ensure that the quantity and type of quality assurance (QA) samples collected meet the requirements of the work plans.

4.0 Project Planning

This section provides a list of general equipment used during rock core preparation and various health and safety considerations.

4.1 General Equipment

- Pocket penetrometer (Gilson or equivalent)
- Indelible black ink pens and markers
- Appropriate sample containers
- Box cutter
- Labels and appropriate forms/documentation for sample shipment
- Nitrile or appropriate gloves
- Sample chain-of-custody forms
- Personal protective clothing and equipment
- Teflon®-lined spatulas and pans and knives, trays, bowls, trowels, or spoons
- Decontamination supplies

Unconfined Compressive Strength (Pocket Penetrometer)

Project-Specific SOP 1-5

Revision: 1

Date: January 17, 2017

5.0 Pocket Penetrometer Procedure

After sufficient curing time (approximately 7 days), the samples will be measured for unconfined compressive strength.

Measurement procedures are described below:

1. Move the ring toward the handle to the lowest reading on the scale. The ring should rest against the lower edge of the instrument handle.
2. Hold the top portion of the handle and slowly push the piston into the soil up to the calibration groove located ¼" from the tip.
3. Read the unconfined compression strength directly in tons per square foot (tons/ft²) or in kilograms per square centimeter (kilograms/cm²) on the low load side of ring closest to top of handle. Convert to pounds per square inch (psi): 1.0 tons/ft² = 13.89 psi.
4. Record the reading in a bound laboratory notebook.
5. For very soft soils, attach the adaptor foot. To use the penetrometer with the adaptor, grip the handle and push the foot into the soil up to the full thickness of the adapter. The reading should be divided by 16 to obtain the correct unconfined compressive strength of the soil.
6. Values above 50 psi are considered acceptable.

6.0 Equipment Cleaning/Decontamination

Disposable equipment will be used when available. In addition, plastic, Teflon, epoxy-coated, or Teflon-coated equipment will be used to prevent mercury amalgamation with metal components. To ensure that samples are not contaminated by equipment or containers, it is necessary to follow certain procedures for cleaning or decontaminating equipment. All equipment in direct contact with the sample must be cleaned between each sample. Decontamination procedures for this pocket penetrometer include:

1. Wash the penetrometer with phosphate detergent and a stiff brush, being sure to remove gross contamination.
1. Rinse all surfaces of the equipment with de-ionized or distilled water.
2. Using a spray bottle, apply a layer of phosphate-free detergent to all surfaces.
3. Vigorously scrub all surfaces of equipment.
4. Rinse all surfaces again with de-ionized or distilled water until all detergent has been removed.
5. Place the equipment in the drying rack.

7.0 Quality Control

Quality control procedures will consist of the multiple measurements at different locations on the molded sample. The precision of each measurement will be determined by calculating the relative percent difference (RPD) between original measurement and the duplicate measurement. The RPD should be less than +/- 35 percent. RPD is calculated as follows:

$$RPD = \frac{[(A-B)/A+B]}{2} \times 100$$

Where: A = Original sample result
B = duplicate sample result

8.0 Documentation

Bound laboratory logbooks shall be used for the maintenance of laboratory records. All aspects of sample preparation, such as sample observations, deviations to procedures, laboratory duplicate precision, decontamination, and calibrations, shall be documented in the laboratory logbooks. The logs documenting the preparation sequence shall be documented daily.

All entries in laboratory logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's activities. Corrections to logbook entries will be accomplished by a single cross out with the date and initials of the person making the entry. White out or correction tape is not permitted. tape is not permitted.

Appendix F

Appendix F

Reactive Sulfide Additive Comparison

A comparison of reactive sulfides was completed (Table 1) to aid in the selection of stabilization additives to be tested. The comparison was divided into two categories as follows:

- Generic chemicals
 - Calcium Polysulfide
 - Sodium Sulfide
- Proprietary chemicals
 - FerroBlack® (with and without the addition of Calcium Oxide)
 - Molecular Bonding System (MBS®) chemicals

The properties that were compared include demonstrated use and effectiveness, implementability, and cost (both relative and total). Demonstrated use and effectiveness was determined based on previously documented successful use of the reactive sulfide during bench, pilot, and full scale applications. Implementability included health and safety issues based on health and safety warnings, physical and chemical properties, and handling and delivery considerations of the chemical. Total chemical cost was determined using the unit cost provided by the vendors and calculating the possible range of quantities required. The range of chemical quantities required was calculated using the total mass of soil to be treated and the range of additive concentrations that will be tested; for example, calcium polysulfide will be tested at the bench scale in the range of 1.5-10 weight percent (weight percent based on weight of additive relative to weight of soil). Relative chemical cost (per kilogram of mercury to be treated) was determined using the total chemical cost and assuming a concentration of 1500 and 6000 milligrams of mercury per kilogram of soil.

Utilizing this comparison (Table F-1), calcium polysulfide was selected as the generic chemical to be tested. The relative cost for calcium polysulfide was \$3.08 to \$20.53 per kg of Hg (total cost ranged from \$133,560-890,400). The relative cost of sodium sulfide was \$5.50 to \$26.67 per kg of Hg (total cost ranged from \$238,170-1,587,830). Calcium polysulfide also has a greater demonstrated use and effectiveness at pilot and full scale testing. Similar implementability and health and safety issues will be encountered with both generic chemicals.

Comparing the proprietary chemicals was not as definitive because of a lack of information regarding the products. Both chemicals had similar costs and implementability issues. The relative cost of FerroBlack® and calcium oxide was \$11.44 (without calcium oxide) to \$94.60 (with calcium oxide) per kg of Hg (total cost of \$495,400-4,096,595), while the MBS® relative cost was \$22.00 to \$73.33 per kg of Hg (total cost of \$952,700-3,175,660). Due to a higher availability of chemical specifications and knowledge of previous successful use and effectiveness, FerroBlack® was selected as the proprietary chemical to be tested. FerroBlack® will be tested both with and without the addition of calcium oxide, as suggested by REDOX Solutions, LLC.

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Table F-1. Additive Comparison
Bench Scale Treatability Study Work Plan
LCP Chemicals, Inc. Superfund Site
Linden, Union County, New Jersey

Stabilization Chemical	Generic Chemicals		Proprietary Chemicals		
	Calcium Polysulfide	Sodium Sulfide	Ferrous Sulfide (FerroBlack®)	Ferrous Sulfide (FerroBlack®) and Calcium Oxide	Molecular Bonding System (MBS®)
Demonstrated Effectiveness	<ul style="list-style-type: none"> Demonstrated effective at bench, pilot and full scale testing 	<ul style="list-style-type: none"> Demonstrated effective at bench scale, and in conjunction with calcium polysulfide at pilot and full scale testing 	<ul style="list-style-type: none"> Demonstrated effective at bench scale testing and field pilot and large scale applications 	<ul style="list-style-type: none"> Demonstrated effective at bench scale testing 	<ul style="list-style-type: none"> Demonstrated effective at bench scale and ex-situ testing
Health and Safety	<ul style="list-style-type: none"> High pH Corrosive Strong odor Potential generation of H₂S gas 	<ul style="list-style-type: none"> Corrosive Prevent contact with acids Maintain at high pH Potential generation of H₂S gas 	<ul style="list-style-type: none"> Contains sulfide Prevent contact with acids Maintain at high pH 	<ul style="list-style-type: none"> Contains sulfide Prevent contact with acids Maintain at elevated pH Monitor Hg emissions with application of calcium oxide 	<ul style="list-style-type: none"> Proprietary mixture, no health and safety issues known
Physical and Chemical Properties	<ul style="list-style-type: none"> Thick orange liquid pH of 10.5 	<ul style="list-style-type: none"> Liquid-phase (60% solid dissolved in water) 	<ul style="list-style-type: none"> Solid mass suspended in liquid (10 micron sized particles of FeS) 	<ul style="list-style-type: none"> FerroBlack® - solid mass suspended in liquid (10 micron sized particles of FeS) Calcium oxide - solid 	<ul style="list-style-type: none"> Solid-phase Can be wetted to ensure mixing
Unit Cost	Graus Chemicals, Remotox bulk \$1.484/gal ¹	Sodium Sulfide \$500/ton ²	REDOX Solutions LLC, FerroBlack® bulk \$0.39/lb ¹	REDOX Solutions LLC, FerroBlack® bulk \$0.39/lb ¹ Calcium Oxide \$120/ton ²	Solucorp Industries Ltd, MBS® \$30-100/waste ton ¹ (generated amount of stabilized material)
Quantity Required* (Assuming 23,600 yds ³ of soil to be treated)	Calcium Polysulfide (29%) <ul style="list-style-type: none"> minium 476 tons for 1.5% (w/w) maximum 3176 tons for 10% (w/w) 	Sodium Sulfide (60%) <ul style="list-style-type: none"> minimum 476 tons for 1.5% (w/w) maximum 3176 tons for 10% (w/w) 	FerroBlack® <ul style="list-style-type: none"> minimum 635 tons for 2% (w/w) maximum 4764 tons for 15% (w/w) 	FerroBlack® <ul style="list-style-type: none"> minimum 635 tons for 2% (w/w) maximum 4764 tons for 15% (w/w) Calcium Oxide <ul style="list-style-type: none"> 3175 tons for 10% (w/w) 	Assume 31,800 waste tons of treated soil (assuming density of 1.6 g/cm ³)
Total Chemical Cost*	<ul style="list-style-type: none"> minimum \$133,560 maximum \$890,400 	<ul style="list-style-type: none"> minimum \$238,170 maximum \$1,587,830 	<ul style="list-style-type: none"> minimum \$495,400 maximum \$3,715,920 	<ul style="list-style-type: none"> minimum \$876,480 maximum \$4,096,595 	<ul style="list-style-type: none"> minimum \$952,700 (\$30/waste ton) maximum \$3,175,660 (\$100/waste ton)
Relative Cost/kg of Hg* (assuming Hg 1500 mg/kg soil)	<ul style="list-style-type: none"> minimum \$3.08 maximum \$20.53 	<ul style="list-style-type: none"> minimum \$5.50 maximum \$36.67 	<ul style="list-style-type: none"> minimum \$11.44 maximum \$85.80 	<ul style="list-style-type: none"> minimum \$20.24 maximum \$94.60 	<ul style="list-style-type: none"> minimum \$22.00 maximum \$73.33
Relative Cost/kg of Hg* (assuming Hg 6000 mg/kg soil)	<ul style="list-style-type: none"> minimum \$0.77 maximum \$5.13 	<ul style="list-style-type: none"> minimum \$1.38 maximum \$36.67 	<ul style="list-style-type: none"> minimum \$2.86 maximum \$21.45 	<ul style="list-style-type: none"> minimum \$5.06 maximum \$23.65 	<ul style="list-style-type: none"> minimum \$5.50 maximum \$18.33
Handling and Delivery Considerations	Difficult <ul style="list-style-type: none"> Strong odors Caustic 	Difficult <ul style="list-style-type: none"> Caustic Challenges with mixing and slurry generation High pH must be maintained 	Medium <ul style="list-style-type: none"> Addition of FerroBlack®, which is solid particles suspended in liquid 	Medium <ul style="list-style-type: none"> Recommended application of Calcium oxide first, monitor until a temperature of 90 degrees Fahrenheit is reached Followed by addition of FerroBlack®, which is solid particles suspended in liquid 	Easy <ul style="list-style-type: none"> Mixing of non-hazardous solid material Can add water to aid in mixing

* Range in quantities based on range of concentrations of additives to be tested in bench scale treatability study work plan

¹ Price quote provided by commercial supplier listed

² Price quoted from internet search